

**U.S. DEPARTMENT OF COMMERCE
National Technical Information Service**

PB-256 364

**Extension of Carbon-Hydrogen Method
to Include Determination of Volatiles
or Loss on Ignition (L.O.I.) at 950 C**

Environmental Protection Agency

1971

PB 256 364

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**NATIONAL TECHNICAL
INFORMATION SERVICE**
U. S. DEPARTMENT OF COMMERCE
SPRINGFIELD, VA. 22161

BIBLIOGRAPHIC DATA SHEET	1. Report No. SWR-156	2.	3. Recipient's Accession No. PB-256 364
	4. Title and Subtitle Extension of Carbon-Hydrogen Method to Include Determination of Volatiles or Loss on Ignition (L.O.I.) at 950 C		5. Report Date 1971
7. Author(s) Donald L. Wilson		8. Performing Organization Rept. No.	
9. Performing Organization Name and Address Environmental Protection Agency Cincinnati, Ohio Solid Waste Research		10. Project/Task/Work Unit No.	
		11. Contract/Grant No.	
12. Sponsoring Organization Name and Address		13. Type of Report & Period Covered	
		14.	
15. Supplementary Notes			
16. Abstracts The following topics are discussed: apparatus, reagents, safety precautions, sample preparation, procedure, standarization, calculations, method evaluation.			
17. Key Words and Document Analysis. 17a. Descriptors *Solid wastes, *Chemical analysis *Oxygen, Laboratory equipment, Experimental design, Ignition, Combustion, Volatility, Safety. 17b. Identifiers/Open-Ended Terms *Carboy hydrogen analysis.			
17c. COSATI Field/Group 13 B 7 C 14 B			
18. Availability Statement National Technical Information Service Springfield, Virginia 22161		19. Security Class (This Report) UNCLASSIFIED	21. No. of Pages 14p
		20. Security Class (This Page) UNCLASSIFIED	22. Price 3.50/2.25

EXTENSION OF CARBON-HYDROGEN METHOD
TO INCLUDE DETERMINATION OF VOLATILES OR
LOSS ON IGNITION (L.O.I.) AT 950 C

A Solid Waste Research
Open-File Report (RS-03-68-17)

written by
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U.S. ENVIRONMENTAL PROTECTION AGENCY
Office of Research and Monitoring
Solid Waste Research
1971

FOREWORD

In publishing an analytical method which employs specific brands of laboratory supplies and instrumentation, the U.S. Environmental Protection Agency does not imply that other commercial products could not be utilized with appropriate modifications in the procedure.

This method is meant to be used in conjunction with the "Laboratory Procedure for the Gravimetric Determination of Carbon and Hydrogen in Solid Wastes" and was written as such.

→ the following topics are discussed:

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DISCUSSION

The oxygen content of solid wastes samples is found indirectly from the volatile analysis.¹ However, the commonly used method^{2,3} of determining volatiles for solid wastes samples presents an awkward means of finding the oxygen content. The standard volatile-ash method for solid wastes samples involves the heating of the sample to 600 C, but, at this temperature, the oxygen combined in carbonates is partially lost due to decomposition of carbonates. Therefore, additional analyses⁴, which determine carbonate content of the samples and ash after volatilizing, must be performed to determine the degree of decomposition of carbonates during volatilization.

A much easier approach to finding the oxygen content of solid wastes samples is to volatilize the samples at a temperature at which almost all of the carbonates decompose.

Although the temperature in the regular volatile-ash method can be raised, a carbon-hydrogen method⁵ for solid wastes samples provides a quick and easy means of determining volatiles at 950 C, a temperature at which most carbonates, especially the most commonly present calcium carbonate, are decomposed.

The carbon-hydrogen method requires that a combustion aid (iron chips) be added to the sample. This combustion aid oxidizes and increases in weight during the carbon-hydrogen analyses. The percent weight increase

due to oxidation must be determined for each new batch (usually 5 pounds) of iron chips. The increase in weight of the iron chips is then subtracted from the weight of the ash remaining after the carbon-hydrogen analyses in order to obtain the true weight of the material volatilized.

In the carbon-hydrogen procedure, samples are retained in clay or nickel boats. Since nickel boats may undergo weight changes during the carbon-hydrogen tests, only clay boats may be used in this procedure.

The terms volatile and ash are commonly used in solid waste management; however, they are often misunderstood and misleading. A more meaningful term, which will be used in this method, is loss-on-ignition (L.O.I.) at a particular temperature, in this procedure, 950 C.

APPARATUS

The apparatus for this method is no different than those described in the procedure for carbon-hydrogen determinations; therefore, the list of apparatus is not repeated here. The only exception to this is that only clay (not nickel) combustion boats are used to retain the samples.

REAGENTS

The chemical requirements and the preparation of reagents for this method are no different than those described in the procedure for carbon-hydrogen determinations; therefore, the information is not repeated here.

SAFETY PRECAUTIONS

The safety precautions are the same as those outlined in the carbon-hydrogen method. No additional hazards are involved in this method.

SAMPLE PREPARATION

The techniques of sample preparation for this extension of the carbon-hydrogen method are the same as those mentioned in the basic method and are not repeated here.

PROCEDURE

This procedure applies to all solid waste materials which are analyzed for their carbon-hydrogen contents. Since the mechanics for this procedure are about the same as those outlined in the procedure for carbon-hydrogen contents, only the details pertinent to the L.O.I. @ 950 C analysis only are discussed here.

<u>Procedure</u>	<u>Comments</u>
1. Transfer, at least, 0.2 to 0.5 grams of <u>dried</u> iron chips into each of ten previously ignited, dry clay combustion boats. Determine and record the total weight of each sample plus boat to the nearest 0.0001 gram.	1. a) This weight represents the normal amount of combustion aid used in the carbon-hydrogen method. b) Minimize handling of boats to prevent contamination. c) Lids to boats are not needed.

Procedure

Comments

2. Store each boat, containing a sample, in a desiccator until it is transferred to the combustion tube.
3. Analyze by the carbon-hydrogen method for blanks, except put each boat with ash in a desiccator after the analysis.

NOTE: Ash from samples high in carbonates, may easily absorb moisture from the atmosphere.

d) Keeping the boats in a particular order will prevent mix-up of samples.

e) Boats should be kept in a desiccator until used.

f) As needed, dry a small amount of iron chips at 105 C and store in a screw top bottle.

2. It is convenient to use a stiff asbestos pad to support the boats while in the desiccator and during transfer from one place to another.
3. a) Develop a technique of handling a boat after the carbon-hydrogen analysis.
b) Care must be exercised not to spill the ash content of each boat.
c) The carbon-hydrogen data obtained here may be used as blank values for the carbon-hydrogen analyses.

<u>Procedure</u>	<u>Comments</u>
4. After each boat has cooled to room temperature, determine and record the weight of each boat plus contents to nearest 0.0001 gram.	4. Data is now available for calculating the percent weight increase of iron chips. (See Calculations.)
5. Analyze solid wastes samples for their carbon-hydrogen contents by the regular method, except for each sample: a) determine and record the weight of iron chips used to the nearest 0.0001 gram; b) after each analysis, as above, determine and record the weight of each boat plus contents to the nearest 0.0001 gram. NOTE: Use only <u>clay</u> boats.	5. a) Having the weight of sample plus (clay) boat, weight of iron chips may be determined by re-weighing boat plus contents after iron chips are added to and mixed with the sample. b. Data is now available for calculating L.O.I. at 950 C for each sample analyzed. (See calculations).

STANDARDIZATION

Standards are analyzed in the manner described under "Procedure" in the method for carbon and hydrogen contents. L.O.I. at 950 C standards may be

established by repeated analysis of various solid wastes samples or by preparing a sample containing a definite amount of an inert material like pulverized ceramics.

CALCULATIONS

Formula for percent weight increase of iron chips:

$$W_i = \frac{(A-B)(100)}{B-X}$$

Where: W_i = percent weight increase of iron chips

A = total weight of each boat plus iron chips after combustion in the carbon-hydrogen method

B = total weight of each boat plus iron chips before combustion in the carbon-hydrogen method

X = weight of each boat

(B-X) = weight of iron chips (before combustion)

NOTE: The percent weight increase for one case of ten samples averaged 21.8% and ranged from 17.7% to 24.6%.

Formula for loss-on-ignition (L.O.I.) @ 950 C:

$$\% \text{ L.O.I. @ 950 C} = \frac{[(D-A) + (C)(W_i)](100)}{D-B}$$

Where: D = total weight of each boat, sample and iron chips before combustion in the carbon-hydrogen method

A = total weight of each boat plus contents after combustion in the carbon-hydrogen method

C = weight of iron chips (before combustion) used with each sample

NOTE: All data is on a dry basis.

METHOD EVALUATION

The data from this method is comparable with data obtained by muffling a solid waste sample at 950 C for one hour in air.⁶ Generally, replicates show better agreement with this method than the muffling method, probably because of the greater temperature control in this method.

Once the percent weight increase of the iron chips has been established, this method adds about 5 to 10 minutes extra time to each carbon-hydrogen test.

Metals not removed during sample preparation could interfere with the accuracy of this analysis. The L.O.I. @ 950 C could even be less than that at 600 C since more metals could oxidize at the higher temperature and with the pure oxygen atmosphere.

ACKNOWLEDGMENT

The author thanks James U. Doerger for performing the laboratory tests necessary to establish this method.

REFERENCES

1. Wilson, Donald L. Procedure for the Mathematical Determination of Total Oxygen in Solid Wastes. Solid Waste Management Office. (In preparation.)
2. American Public Works Association. Test for Volatile Solids and Ash. In Municipal refuse disposal. 3d. ed. Chicago, Public Administration Service, 1970. p.393-395.
3. Ulmer, Nancy S. Laboratory Procedure for the Determination of Volatiles in Solid Wastes. (For Method's Manual). Solid Waste Management Office. (In preparation.)
4. Wilson, Donald L. Laboratory Procedure for the Gravimetric Determination of Carbonate Carbon in Solid Wastes. Solid Waste Management Office. 1971.
5. Wilson, Donald L. Laboratory Procedure for the Gravimetric Determination of Carbon and Hydrogen in Solid Wastes. Solid Waste Management Office. 1970.
6. Wilson, Donald L. Formulas (Incorporating Decomposition of Carbonates at 600 C) for the Determination of Total Oxygen in Solid Wastes. Solid Waste Management Office. (In preparation.)
7. Cohen, Israel R. Laboratory Procedures for the Preparation of Solid Waste Related Materials for Analysis. Solid Waste Management Office. (In preparation.)